

[54] COMBINED THERMAL ANALYZER AND X-RAY DIFFRACTOMETER

[75] Inventors: Timothy G. Fawcett; William C. Harris, Jr.; Robert A. Newman; Lawrence F. Whiting; Frank J. Knoll, all of Midland, Mich.

[73] Assignee: The Dow Chemical Company, Midland, Mich.

[21] Appl. No.: 105,769

[22] Filed: Oct. 6, 1987

Related U.S. Application Data

[63] Continuation of Ser. No. 938,682, Dec. 5, 1986.

[51] Int. Cl.⁴ G01N 23/20

[52] U.S. Cl. 378/80; 378/71

[58] Field of Search 378/70, 71, 80; 374/31, 374/33, 34

References Cited

U.S. PATENT DOCUMENTS

3,051,834 8/1962 Shimula et al. 378/80
 3,263,484 8/1966 Watson et al. 73/15
 3,337,731 8/1967 Kinznetsov et al. 378/80
 4,263,510 4/1981 Ciccarelli et al. 378/80

FOREIGN PATENT DOCUMENTS

923025 4/1963 United Kingdom .
 936910 9/1963 United Kingdom .
 945788 1/1964 United Kingdom .
 1432826 4/1976 United Kingdom .
 2079465A 1/1982 United Kingdom .

OTHER PUBLICATIONS

"Purity Determination by Simultaneous DSC-Thermomicroscopy", by Wiedemann et al.

"Simultaneous . . . of Melting and Freezing Processes", by Van Ters et al., Mettler Instrument A.G.

"Simultaneous Differential Scanning Calorimetry and Small Angle X-Ray Scattering", by Russell et al., Journal of Polymer Science Polymer Phys. Ed., vol. 23 (1985).

Thermochimica Acta, vol. 7 (1973), pp. 131-149, H. G. Wiedemann, "Thermal Analysis and Synthesis of Pentazinc Hexahydroxide Dicarboxate-Investigations by

Thermogravimetry, Thermo Molecular Beam Analysis and X-Ray Measurements".

Analytical Chemistry, vol. 266 (1973), pp. 97-109, H. D. Wiedemann and G. Bayer, "Investigation of Minerals and of Lunar Samples (14163, 14258) by Simultaneous Thermal and X-Ray Analysis".

Journal of Physics E, vol. 7, No. 7 (1974), pp. 509-510, N. Gerard, "Coupling of Thermogravimetric and X-Ray Diffraction Methods".

Thermochimica Acta, vol. 59, No. 3 (1982), pp. 343-359, P. J. Haines and G. A. Skinner, "Simultaneous Differential Scanning Calorimetry and Reflected Light Intensity Measurement".

Advances in X-Ray Analysis, vol. 22, Plenum Press, New York and London (1979), pp. 255-265.

H. E. Göbel, "A New Method for Fast XRPD Using a Position Sensitive Detector".

(List continued on next page.)

Primary Examiner—Craig E. Church

Assistant Examiner—John C. Freeman

Attorney, Agent, or Firm—Burke M. Halldorson

[57]

ABSTRACT

Scientific apparatus and a method are described for observing simultaneously both structural and thermodynamic properties of materials. An X-ray diffractometer and a thermal analyzer are mounted to cooperate and coact on the same sample and to complete a meaningful analysis in a very few minutes. The diffractometer is equipped with a rapid position-sensitive detector connected to a multichannel analyzer to record and display X-ray diffraction data from the sample over an angle of 20° (two theta) or more. The thermal analyzer is preferably a differential scanning calorimeter. By correlating X-ray diffraction and thermal data taken simultaneously while the sample is passing through a range of temperatures and/or environments, structural changes corresponding to thermal events can be identified and elucidated.

9 Claims, 14 Drawing Sheets

